

PATENT

Attorney Docket No. 08009.0010-00

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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|-----------------------------|---|------------------------|
| In re Application of: |) | |
| |) | |
| Katsuya KASE et al. |) | Group Art Unit: 1745 |
| |) | |
| Application No.: 10/781,624 |) | Examiner: K. O'Neill |
| |) | |
| Filed: February 20, 2004 |) | Confirmation No.: 4112 |
| |) | |
| For: ACTIVE MATERIAL FOR |) | |
| POSITIVE ELECTRODE IN NON- |) | |
| AQUEOUS ELECTROLYTE |) | |
| SECONDARY BATTERY |) | |

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER 37 C.F.R. § 1.132

I, Katsuya KASE, do hereby make the following declaration:

1. I am a Japanese Citizen, residing at 17-3, Isoura-cho, Niihama-shi, Ehime-ken, Japan.
2. I have been awarded a Master of Science from Chiba University, Japan
3. I have been employed by Sumitomo Metal Mining Co., and its predecessor corporations since April 1, 1992, and I am presently Manger of Technical Section, Battery Material Department. During my employment at Sumitomo Metal Mining Co., I have been with Advanced Material Division, Isoura Plant.
4. I have read and understand the rejections presented in the Final Office Action mailed March 27, 2007, in U.S. Patent Application No. 10/781,624, and the reference cited therein, Japanese Publication No. JP 2000-021402.

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5. In particular, I have read Japanese Publication No. JP 2000-021402. In my opinion, a person of ordinary skill in the art of active materials for secondary batteries would recognize that this document does not a lithium composite oxide that inherently possesses a lithium occupancy rate of 98% or more, or a carbon amount of 0.12 weight % or less.

6. Given my education and experience, particularly in the area of battery compositions and manufacturing, I consider myself able to provide the following testimony based on the following experiments conducted by me or under my supervision.

7. Three compositions, A, B, and C, were manufactured in accordance with the examples in Japanese Publication No. JP 2000-21402. In particular, these compositions were manufactured as follows:

8. A mixed solution of nickel sulfate and cobalt sulfate (nickel concentration: 1.45 mol/L; cobalt concentration: 0.27 mol/L), 150g/L caustic soda solution, and 25% aqueous ammonia were dripped while stirring into a reaction tank, forming a composite nickel hydroxide. Suction filtration of the composite nickel hydroxide was then performed. After suction filtration, 0.5 kg, 0.2 kg, or 0.1 kg, of caustic soda was added per 1 kg of hydroxide substance. The mixture was stirred for 5 hours and washed in alkali. Afterwards, the resultant was washed and washed with water until the pH of the filtered water was 9.0 or less. 4 weight % (relative to the nickel hydroxide) of sodium aluminate and pure water was then added (to the respective samples A, B, and C), to make a 0.5 kg/L slurry. the slurry was neutralized to pH 9.5 with dilute sulphuric acid. Aluminum hydroxide covered the surface of the nickel hydroxide. The resultant was

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then dried for 48 hours at 100 °C to form a composite nickel hydroxide for use in a lithium metal composite oxide.

9. The compositions of the composite nickel hydroxides that were made are as follows:

| Sample | Amount of Caustic Soda Per 1 kg of Hydroxides | Amount of SO ₄ ions in obtained Nickel Hydroxides |
|--------|--|---|
| A | 0.5 kg | 0.5 wt % |
| B | 0.2 kg | 0.8 wt % |
| C | 0.1 kg | 1.2 wt % |

10. To 100 g of each of the composite nickel hydroxides, 41.8 g of commercially sold lithium hydroxide anhydrous salt powder was mixed to provide a mixture. Each mixture was then sintered in an oxygen airflow for 4 hours at 250 °C, 9 hours at 450 °C, and 22 hours at 730 °C. A sintered lithium composite oxide body for each sample was obtained, and allowed to cool to room temperature.

11. Each sintered body was crushed in a nitrogen atmosphere using a pin mill. The crushed sintered body was sifted with a 25 µm ultrasonic vibrator, and particles having a diameter of 25 µm or more were removed. The retained powder was further classified by a wind power classifier, and particles having a diameter of 1 µm or less were removed.. The resultant classified lithium composite oxide powder was then dried in a vacuum at 150 °C for 24 hours.

12. X-Ray diffraction analysis was performed on the obtained lithium-metal composite oxide powder. This analysis confirmed the presence of a lithium composite oxide having a hexagonal layered structure. From the X-ray diffraction chart and

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Reitveld analysis, the lithium site occupancy rate for these samples was determined to be 93.1% to 95.1 %.

13. The carbon content of the obtained lithium-metal composite oxide powders were also measured using the high-frequency heating-infrared absorption technique. The measured carbon content ranged from 0.20 to 0.32 weight %.

14. The results of the above measurements are reported in the following tables:

| Sample | Amount of SO ₄ Ions in Nickel Hydroxides as Raw Material | Amount of SO ₄ Ions in obtained Lithium-Nickel Composite Oxides |
|--------|---|--|
| A | 0.5 wt % | 0.6 wt % |
| B | 0.8 wt % | 0.9 wt % |
| C | 1.2 wt % | 1.5 wt 5 |

| Sample | Li Occupancy Rate of Obtained Lithium-Nickel Composite Oxide | C amount of Obtained Lithium-Nickel Composite Oxide |
|--------|--|---|
| A | 95.1 % | 0.20 wt % |
| B | 94.5 % | 0.22 wt % |
| C | 93.1 % | 0.32 wt % |

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15. As shown above, samples A, B, and C, which were manufactured according to the disclosure and examples of JP 2000-21402, exhibit a Li site occupancy rate significantly less than 98%, and a carbon content significantly greater than 0.12 weight %

16. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: sep. 19, 2007

By: Katsuya Kase
Katsuya KASE